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ARPA Coupling Program on Stress-Corrosion Cracking

(Fifth Quarterly Report)

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ABSTRACT

A progress report of the research investigations being carried out on the problem of stress-corrosion cracking of high strength materials under ARPA Order 878 is presented. Work at Carnegie-Mellon University, Lehigh University, Georgia Institute of Technology, The Boeing Company, and the Naval Research Laboratory concerning physical metallurgy, surface chemistry, fracture mechanics, and characterization tests and translation related to stress-corrosion cracking is described. The materials being studied include high strength steels, titanium alloys, and aluminum alloys. Abstracts of recently published reports and a diary of events are included.

STATUS

This is a progress report; work is continuing.

AUTHORIZATION

NRL Problems 61M04-08
63M04-08A
63M04-08C
ARPA Order 878 and
RR 007-08-44-5512

INTRODUCTION

In order to learn how to improve high-strength structural alloys with respect to their resistance to stress-corrosion cracking (SCC) the Advanced Research Projects Agency of the Department of Defense has established under ARPA Order 878 a broadly based interdisciplinary attack upon the problem of SCC in high strength titanium alloys, steels, and aluminum alloys. The project is composed of sectors located in The Boeing Company, Carnegie-Mellon University, Lehigh University, Georgia Institute of Technology, and the Naval Research Laboratory. In addition to having its own research activity, NRL has the responsibility for keeping the entire technical program attuned to DoD needs.

The complex phenomenon of SCC can be divided into four elements as follows: (1) the stress field, (2) the metallic phase, (3) the corrodent phase, and (4) the interface (with or without corrosion-product films) between metal and corrodent. Because of the obvious complexities of the phenomenon (and perhaps additional complexities not yet obvious), an interdisciplinary approach is essential. Scientists prominent in the fields of surface physics and surface chemistry have been enlisted in the project in order to bring the tools of these new sub-disciplines to complement those of traditional electro-chemistry/thermodynamics in order to design new advances in the practical problem.

The reporting system is as follows: Quarterly (commencing 1 January 1967) submissions from each unit of the project are submitted to section editors, who in turn submit the edited sections to NRL for publication as an NRL report. These sections must be kept brief to be manageable, and the project personnel are enjoined to publish the research details in the standard technical journals as a means of most effectively injecting the output of the program into the technological mainstream. When such publications are submitted to a technical journal, the abstract is included in the quarterly report, so that interested readers may contact the author if the subject matter is of immediate interest. Additionally, the abstract will again be published in the quarterly report when the paper finally is printed in the technical journal and is presumably available in reprint form. Reprints or requests for advance copies of such papers (or advance information contained in the papers) should be addressed to the individual author or authors. Commencing with the Third

Quarterly Report selected abstracts of reports and journal articles from outside the ARPA Program in the field of stress-corrosion cracking will be included as a service to readers of the Quarterly series.

The individuals responsible for directing this research at the various institutions and their participation as technical editors for the subject areas of their specialization in these progress reports are as follows:

Carnegie-Mellon University

H. W. Paxton, Project Director
R. P. M. Procter, Section Editor of Physical Metallurgy

Lehigh University

P. C. Paris, Project Director
A. C. Zettlemoyer, Director, Surface Chemistry Center
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Georgia Institute of Technology

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The Boeing Company

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Naval Research Laboratory

B. F. Brown, Program Director
E. P. Dahlberg, Assistant (General Editor)
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A. PHYSICAL METALLURGY

STEELS

Carnegie-Mellon University

Steel sheets containing different carbon contents (covering the range 0.03 to 1.46% C) but with matched composition with respect to other elements present have been obtained from the U. S. Steel Research Laboratories; the prior processing history of these steels is known. These sheets will be used as specimens in the investigation of the diffusion of hydrogen in steel at room temperature which is the overall objective of this project. The experimental apparatus being used was described on page A-5 of the previous quarterly report in this series. It is intended to investigate hydrogen diffusion through specimens in the "annealed" and "annealed and cold-rolled" conditions. One side of the specimen under investigation is abraded under water under carefully controlled conditions. The other side of the specimen is exposed to the vacuum system (vacuum of the order of 10^{-8} to 5×10^{-8} torr) and the hydrogen diffusing through is measured with the mass spectrometer. An experimental difficulty was encountered in that a hydrogen background level (the source of this hydrogen is at present unknown) in the system built up to appreciable levels so that the small additional amount of hydrogen diffusing through the specimen into the system could not be measured with sufficient accuracy. This problem has been partially overcome by incorporating a glass three-stage fractionating oil-diffusion pump (25 liter/sec. capacity, Consolidated Vacuum Corp., Model GF-26) into the system to remove the excess hydrogen. A Hydrogen Standard Leak (Consolidated Electrodynamics Co. No. 139072) was also temporarily incorporated into the system to enable the mass spectrometer to be calibrated. Further attempts are currently being made to overcome this problem completely and to ensure that the apparatus is functioning with maximum efficiency and reproducibility.

During a routine inspection, a wiring error was located in the potentiostat used to generate the polarization curve shown in fig. A-1 of the Fourth Quarterly Report in this series. This curve must therefore be considered to be of doubtful validity.

Georgia Institute of Technology

Studies of the effects of variations in the major and minor alloying

elements on the stress-corrosion cracking of ALMAR 362 maraging stainless steel are underway. 187 k.s.i. tensile strength and 180 k.s.i. yield strength are reported to be the optimum heat-treated mechanical properties. It has been found that by cold-working prior to aging, tensile and yield strengths in excess of 240 k.s.i. can be achieved. Initial corrosion studies indicate that in this condition the alloy cracks under a cathodic potential of 10 mA/in.^{2} and the fracture appears to be due to hydrogen-embrittlement. A more complete analysis of this alloy is underway. Studies of environmentally-induced crack-growth of several other steels, held at stresses above the yield point, are also underway. Positive and negative potentials are being applied to a series of these specimens.

ALUMINUM ALLOYS

Carnegie-Mellon University

The corrosion behavior of an Al-4% Cu alloy in a 3.5% NaCl solution is being investigated. Material that has been naturally aged and oxidized in air exhibits non-preferential pitting and exposure for one week produces no degradation in the mechanical properties. Scratching the oxide film causes preferential attack along the line of the scratch. In contrast, material aged at 180° C to maximum strength is preferentially attacked along the grain-boundaries and a significant degradation of the 0.2% proof stress results. However, scratching the surface of these specimens does not promote extensive attack along the scratch lines.

The investigation into the relationship between mechanical properties and microstructure is continuing with the Al-Zn-Mg system. Heat treatments designed to vary the size of the precipitate-depleted zone adjacent to the grain-boundaries have as yet shown no variation in the mechanical properties, derived from tensile tests, directly attributable to the width of the depleted zone. These specimens are shortly to be tested under stress-corrosion conditions.

Lehigh University

Immersible crack-opening displacement (COD) gauges have been prepared and compliance calibration has been performed on aluminum (7075-T6) and titanium (Ti-6Al-4V) stress-corrosion specimens. The COD method appears to be sensitive enough to measure a crack growth of approximately 0.001 inches. Stress-corrosion crack growth rates

are being measured for Ti-6Al-4V in distilled water and in salt solutions at various temperatures using both the COD method and electrical resistivity methods. For the aluminum alloy, the effect of specimen thickness on stress-corrosion crack propagation is being studied. During this quarter, an investigation into crack-blunting phenomena has been initiated. Stress-corrosion testing of steel alloys above and below the K_{Iscc} is being conducted and the kinetics of crack-blunting will be determined, using metallographic techniques. This study is directed towards elucidating the role of blunting in the arresting of cracks in corrosive environments.

TITANIUM ALLOYS

Carnegie-Mellon University

The importance of interstitial impurities in the dynamics of deformation of titanium has been emphasized in a recent paper (1). These impurities also affect the kinetics of phase transformation in titanium and its alloys (2). Oxygen is one of the more important interstitial impurities in titanium and it is by far the most difficult to remove. As a preliminary to studying both the above affects, several approaches have been used in attempting to obtain high-purity, very low oxygen titanium for experimental purposes. The initial investigation showed that floating-zone zone-refining techniques could only produce oxygen concentrations of the order of 100 to 150 ppm. Subsequent attempts to remove further oxygen from the titanium by equilibrating with a getter of titanium at a different temperature, under high vacuum, were made, but chemical analysis for oxygen showed that no significant purification was obtained. Verhoeven (3) has discussed the possibility of using electrotransport phenomena as a basis for purifying metals. Calculations have suggested that, for oxygen in β-titanium, a purification (expressed as the ratio of oxygen concentration in the purified- and starting-materials) of 0.076 could be expected by this method; (assuming $D_{\beta}^{O_2} \approx 0.45 \times 10^{-5}$ cm^2/sec., and a differential mobility of $U = 1.44$ cm^2/volt. hr., with an electric field of 0.2 volt/cm, a 10 cm long specimen and a time of 10^6 sec.) The possibility of combining electron-beam

(1) H. Conrad, Canad. J. Phys., 45, 1967.
(2) M. K. McQuillan, Met. Rev., 8, 1963.
(3) J. D. Verhoeven, J. of Metals, January 1966.

floating-zone techniques with electrotransport and thermal diffusion phenomena, in order to obtain significant purification of research quantities of material, is now being investigated.

Georgia Institute of Technology

A special furnace designed to achieve optimum grain growth in commercial and special titanium-aluminum alloys is in its final stages of completion. Preliminary studies have indicated that a grain diameter of approximately two to three millimeters may be obtained. In addition to alloys obtained from Reactive Metals Inc. and Titanium Metal Corp., small quantities of titanium-aluminum binary alloys have been prepared, by vacuum-arc melting, at the Georgia Institute of Technology. In considering the fracture of these alloys the plastic zone at the tip of the crack came under consideration. Experiments were designed to examine specimens stressed above the elastic limit, to simulate conditions at the crack tip. In the original tests it was planned to use loads approximately half-way between the yield and tensile strengths. Several specimens from a duplex-annealed 8-1-1 alloy were machined and carefully polished. A series of tensile tests was conducted and an environmental test devised; a plastic cup was fitted to the tensile specimens to hold 3.5% NaCl solution. In each environmental test the specimen broke before the static testing load was achieved. A fracture surface is shown in figure A-1; arrows denote the area where rapid environmentally-induced crack growth had occurred. In figure A-2 the fracture surface is shown at an angle so that the growth on the surface can be seen more clearly than in the first case. Electron fractography was performed on the fracture surfaces and figure A-3 shows the typical cleavage found in environmentally-induced crack-growth regions. This type of testing provides several possibilities for determining what is occurring in plastically stressed regions under environmental attack. Additional studies are now under way. In addition to 8-1-1, other alloys, including 5Al-2.5Sn and 6Al-4V, are being tested. The environmental testing will be conducted with dead-weight loading at specified stress levels above the yield point. Furthermore, positive and negative potentials will be applied to samples utilizing a Beckman Electroscan 30 potentiostat. A specimen designed to produce a specified cross-section and length to which the plastic zone will be confined, is being developed. When additional experimental information is available stress-analysis studies with Lehigh University and the NRL will be initiated.

Fig. A-1

Fig. A-2

Fig. A-1 and A-2 - Macrographs of the fracture surface of a titanium 8-1-1 alloy tensile specimen broken in 3.5% NaCl solution, taken from different angles; x8 magnification.

Fig. A-3 - Typical electron-fractograph of the environmentally-induced crack growth region of the specimen shown in figs. A-1 and A-2; x6,000 magnification.

Field-emission microscopy studies of the surface characteristics of and of adsorption on susceptible and non-susceptible titanium alloys are being undertaken. Work so far has been confined primarily to optimization of the design of the apparatus. Field-ion microscopy studies of the atomic structure of these titanium alloys are also underway. It has been reported in the literature that titanium has been imaged but no information with regard to the imaging gas or potential was provided. After contact with those reporting this work, it was found that only preliminary experiments, which indicated the possibility of imaging titanium in neon and hydrogen, had been performed. No fully developed image had been obtained. Work at Georgia Institute of Technology has now progressed past this preliminary stage and the first successful field-ion microscopy of pure titanium and of titanium alloys is in progress. To date, partial images from these materials have been developed. However, the stability of the images still leaves much to be desired and further technique development is required. The success to date indicates that both titanium and titanium-aluminum alloys can be imaged once the optimum in sample preparation, imaging gas, and voltage control is obtained.

Limited studies of the surface oxide characteristics in the 50A range, using x-ray line-profile analysis, will be undertaken. Single crystals of Ti and α-Ti-4% Al alloy are being prepared for this work.

The Boeing Company

The metallurgical aspects of the stress-corrosion cracking of titanium alloys has been studied. It has been shown that failure of the α-phase in Ti-Al alloys and in the commercial alloy Ti-8Al-1Mo-1V, under stress-corrosion cracking conditions, occurs by cleavage on the $\{10\bar{1}8\}$ - $\{10\bar{1}7\}$ plane of the hexagonal phase. The general phase structure of these alloys has been described and it is shown that the $\alpha \rightarrow \alpha + \alpha_2$ (Ti_3Al) transformation is of considerable importance in determining susceptibility. In the commercial alloy, presence of the β-phase and the martensitic phases reduces susceptibility. Results on the binary Ti-Al alloys have indicated that the onset of susceptibility to stress-corrosion cracking occurs between 4-5% aluminum, which correlates with a change from homogeneous to planar dislocation arrangements. The influence of stress on the nucleation and propagation of a crack has been analyzed for precracked specimens, using the methods of linear elastic mechanics. From these results several factors which influence the nucleation stage may be discussed.

It is proposed that for a propagating crack there are two components of fracture, i.e., electrochemical and mechanical. At low α-phase strength levels or aluminum contents the electrochemical component predominates, while at higher strength levels or aluminum contents the mechanical component increases. Some qualitative correlations have been made between mechanical properties, deformation structures, fracture mode, and susceptibility to stress-corrosion cracking. These results are all reported in greater detail in the Boeing Technical Reports listed in section D of this document.

The following special projects have also been completed at Boeing during the past quarter: (1) A Possible Role of Hydrogen in Stress-Corrosion Cracking of Titanium Alloys: The various mechanisms that have been put forward to explain stress-corrosion cracking have been briefly summarized. The experimental observations on stress-corrosion cracking in titanium alloys in widely different environments have been discussed. Evidence from the literature for and against a positive role of hydrogen in the stress-corrosion cracking of titanium alloys has been documented. An experimental approach has been outlined in an attempt to establish if environmental cracking in titanium alloys in specific environments is due mainly to a stress-corrosion mechanism or to a process similar to hydrogen-embrittlement. (2) The Oxidation Behavior of Titanium: The oxidation characteristics of titanium are reviewed from the kinetic data available in literature. The experimental techniques and the relevant conclusions have been summarized. An experimental approach has been outlined for the preliminary stage of a more comprehensive investigation to delineate the effect of oxygen and ozone on the mechanical and structural properties of titanium alloys that are candidate materials for structural applications.

B. SURFACE CHEMISTRY

ELECTROCHEMISTRY

Naval Research Laboratory

Work on the electrochemical oxidation of hydrogen on pure iron and platinum has given insight into the mechanism of passivation. On a platinum electrode the reduction in the rate of hydrogen oxidation, as the potential becomes more positive, was shown to be due to a fractional coverage of the surface with anions from the solution (hydroxide in sodium hydroxide and sulfate in sulfuric acid solutions). The formation of oxygen films was not the cause of passivity. Similarly, passivity of iron for the hydrogen oxidation reaction is due to adsorption of a partial anionic monolayer. Raising the potential increases the activation energy required for hydrogen oxidation. A base oxide film or adsorbed oxygen atoms may be present on iron. If they are, they are good electronic conductors and are very thin (probably atomic dimensions). The retardation of hydrogen oxidation or passivity is not due to an increase in the thickness of such an oxide or to the formation of another oxide on top of the initial oxide.

MACROSCOPIC SURFACE MEASUREMENTS

Georgia Institute of Technology

The relationships to crack initiation of micromechanical effects and chemical and physical properties of metal and alloy surfaces were examined by electron fractography, autoradiography, and acoustic emission and pulse echo techniques. LEED and associated methods will be used to characterize the surfaces used in the micromechanical experiments and conversely the micromechanical measurements are expected to indicate the alloy systems whose surface and oxidation properties should be investigated. High strength alloys of practical importance as well as single crystals will be studied. During the quarter the major part of a new custom-designed LEED system was received from Varian Associates. It incorporates a reaction chamber which can be isolated from the main system for the study of chemical reactions at high pressures and temperatures. A separate gas handling system for corrosive gases is now being assembled. Eventually the system will

also include a high energy electron diffraction attachment for studying reaction products in the thick film region. Improvements have been made in the electronic circuitry and result in increased sensitivity for detecting impurities by their Auger peaks.

Lehigh University

Electron paramagnetic resonance studies of oxygen interaction with rutile (TiO_2) and zinc oxide are continuing. Various treatments of zinc oxide with nitrogen dioxide, nitric oxide and nitrous oxide as well as with chlorine have been used to help resolve some of the disagreements among those who have reported E.P.R. investigations of the zinc oxide/oxygen system.

Oxygen deficient zinc oxide was produced by outgassing samples at 500° C and 10^{-5} torr for two hours, and gave spectra with a strong signal at g = 1.96 and a weak signal at g = 2.003. Nitrogen dioxide ($\rightleftarrows N_2O_4$) treatment of the non-stoichiometric oxide at room temperature and about 10 torr was shown by mass spectrographic analysis to have left large quantities of nitrogen in the gas phase. Nitrogen dioxide treated zinc oxide gave spectra with a strong signal at g = 2.015 but with only a weak signal at g = 1.96. Similar experiments with nitric oxide only decreased and split the peak at 1.96. Heating the sample above 250° in contact with nitric oxide produced a spectrum (taken at the liquid nitrogen temperature used for all spectral measurements) with a sharp symmetric signal at 2.015. Similar results were observed for nitrous oxide treatment. Adsorption of the nitrogen oxides on precooled (in liquid nitrogen) samples resulted in complex spectra probably due to the gaseous oxides adsorbed on the ZnO. Chlorine adsorbed on outgassed ZnO produced a strong signal at 2.015 identical to that produced by the nitrogen oxides. The signal at g = 2.015 is caused by the removal of an electron from the zinc oxide, possibly from a shallow surface level. The decrease in intensity of the signal at g = 1.96 is due either to oxidation of Zn^+ to Zn^{++}, or to the refilling of the oxygen vacancies. The observed splitting of the signal at 1.96 may depend on the improved resolution which is possible with weaker signals; the signal is likely to be an unresolved overlapping doublet before nitrogen oxide treatment. The above studies and some results obtained with rutile will be reported at the Third Middle Atlantic Regional Meeting of the American Chemical Society on February 1 in Philadelphia.

Naval Research Laboratory

Experiments in a hydrogen effusion apparatus involving growth of a protective film on mild steel in an aqueous medium containing cobalt ions have continued with periodic withdrawal of specimens from the hot (300° C) zone. A specimen withdrawn after 100 days of heating was shown (spectrographically) to contain cobalt in the magnetite film on the metal surface. Hydrogen effusion rates have not so far indicated any striking difference between a cobalt solution and a solution of 15% sodium hydroxide without cobalt. Mild steel tubing has been coated with an oxide film by heating at 115° C and atmospheric pressure in a concentrated sodium hydroxide solution doped with manganese ions. An attempt will be made to weld capsules containing various solutions for effusion experiments on this coating.

Studies of the adsorption and desorption of radiostearic acid on ferrous metals and alloys are continuing. Surfaces of polished nickel and of 304 and 416 alloy steels adsorb less compacted monolayers of stearic acid than does Armco iron as shown by methylene iodide contact angles of 66-67°, compared to 69-70° for Armco iron and 72-73° for soft glass. Extrapolation of the straight-line relationship between contact angle and radioactivity indicates surface coverage of the nickel and alloy steels is about 80% of a compacted monolayer, and of the Armco iron is 95% or more of a compacted monolayer. The present polishing procedure gives surface roughness factors equivalent to that of fire-polished soft glass (assumed equal to unity); the earlier polishing technique gave roughness factors of about 1.3 for Armco iron. Desorption of stearic acid-1-C^{14} at 95° and 105° follows the same pattern for all of the ferrous metals and alloys studied with about 25-30% of the stearic acid being rather firmly bonded to the metal surface.

C. CHARACTERIZATION TESTS AND TRANSLATION

Two additional large-scale tension stress-corrosion cracking tests of the type reported previously (NRL Memorandum Report 1775, page 22) were carried out at NRL on Ti-7Al-2Cb-1Ta alloy. Each specimen was 6 by 1 by 24 in. and contained a surface fatigue crack. Each was tested in a 3.5% NaCl solution under sustained load until failure. The first contained a surface crack 0.4 in. long and 0.18 in. deep. Using ultrasonic techniques, stress-corrosion cracking was first observed at a stress of 57.3 ksi, corresponding to a stress-intensity (K) level of 32.2 ksi$\sqrt{in}$. This correlates very well with the previously determined K_{Iscc} value of 31.5 measured using a cantilever-beam test. The second test specimen contained a flaw 1.04 in. long and 0.32 in. deep. This specimen showed signs of cracking at a stress of 49.5 ksi and failed after 21 min. The K-level corresponding to this stress was 42.6 ksi$\sqrt{in}$. This significant difference from the cantilever-beam result can be mitigated in part by the fact that the crack initially extended by SCC only on one side. Some cracking was observed at the welded specimen-tab joint that might have resulted in nonaxial loading. Thus the K determination might be in error.

A similar test of a Ti-6Al-4V alloy with a K_{Iscc} of 90 ksi$\sqrt{in}$. was not successful because the lower weld joint fractured. Additional tests of this alloy are planned.

Boeing, in conjunction with Materials Research Laboratory (MRL), is conducting a program to study stress-corrosion cracking in steel and aluminum forgings of selected composition using tapered double cantilever-beam specimens. Materials to be studied in this program will be silicon-modified 4340, Maraging 250, and 9Ni-4Co-0.45C (bainitic condition) steels and 7075-T6 aluminum, all supplied as forged billets. Crack growth rates will be determined at different K-levels by subjecting specimens to cyclic as well as sustained loading. The environments will be distilled water and 3.5% NaCl solution. Threshold stress intensity levels (K_{Iscc}) will be determined from K versus crack growth rate curves, i.e., the K-level at which the crack growth rate extrapolates to zero will be considered as K_{Iscc}. These will be compared with K_{Iscc} determined using a "fixed grip" testing technique. In this procedure, the specimen is loaded to a given crack-opening displacement. This displacement is maintained as the crack grows due to stress corrosion, while at the same time the load decreases. This means that K decreases as the crack extends, and it is hypothesized that crack growth will be arrested when K decreases to K_{Iscc}.

All materials have been received by MRL. Clip gauges and systems for holding environment have been designed and tested on 7075-T6 plate. Initially,

difficulty was encountered with salt solution getting under the protective coatings of the clip gauge. Some crack growth (400 microinches per minute) was noted in 7075-T6 plate where the plane of cracking was in the long-transverse grain direction and the applied K-level was 80% of K_{Ic}. However, the aluminum had been electrochemically coupled to the steel loading pins during the test. When the pins were reduced in diameter and wrapped in Teflon, the crack growth rate decreased to 60 microinches per minute. The IR drop across the Teflon was 4 megohms, which indicates a high level of insulation.

The threshold stress-intensity levels (K_{Iscc}) for stress corrosion in 3.5% sodium chloride solution were determined at NRL for nine commercial aluminum alloys. Uniform double-cantilever-beam specimens were used to measure the K_{Iscc} levels. The specimen geometry and test technique were described in the Fourth Quarterly Report of this series (page 26). Figure C-1 shows the K_{Iscc} values as a function of yield strength for several of these alloys. The yield strength for each alloy except one was determined from standard tension tests oriented in the long-transverse grain direction. The alloy 2020-T651 was not tested in this manner, and its yield strength is simply an estimate.

The terminal fracture of titanium alloys containing stress-corrosion cracks has been investigated at Boeing. The length of stress-corrosion cracks was measured in 55 precracked specimens of Ti-6Al-4V and Ti-4Al-3Mo-1V that had previously been sustain-loaded in 3.5% sodium chloride solution. The fracture toughness index, $K_{I\delta}$, of the residual ligament was calculated for each specimen and the data compared with the mean K_{Ic} values that were determined in air. $K_{I\delta}$ is fracture toughness calculated from the length of an environmental crack after failure in a conventional sustained-load SCC test on a fatigue-precracked notched bend specimen. The ratio $K_{I\delta}:K_{Ic}$ was within the range 0.85 to 1.14 except for approximately 10% of the specimens that had higher ratios. The rather wide distribution of values was attributed to the inherent scatter in K_{Ic} and to the irregular shape of many stress-corrosion crack fronts, which prevented the measurement of an effective critical crack length.

The influence of crack length on the $K_{I\delta}:K_{Ic}$ ratio was also examined. This revealed a trend for the $K_{I\delta}:K_{Ic}$ ratio to exceed 1.0 when the crack-length-to-specimen-width ratio was greater than 0.4. This is contrary to the results reported by Brown and Srawley (W. F. Brown and J. E. Srawley, Plane Strain Crack Toughness Testing of High Strength Metallic Materials, ASTM-STP 410, 1967) for specimens containing fatigue cracks of various lengths. In view of the effective crack length problem, the trend should be treated with caution, and requires further investigation.

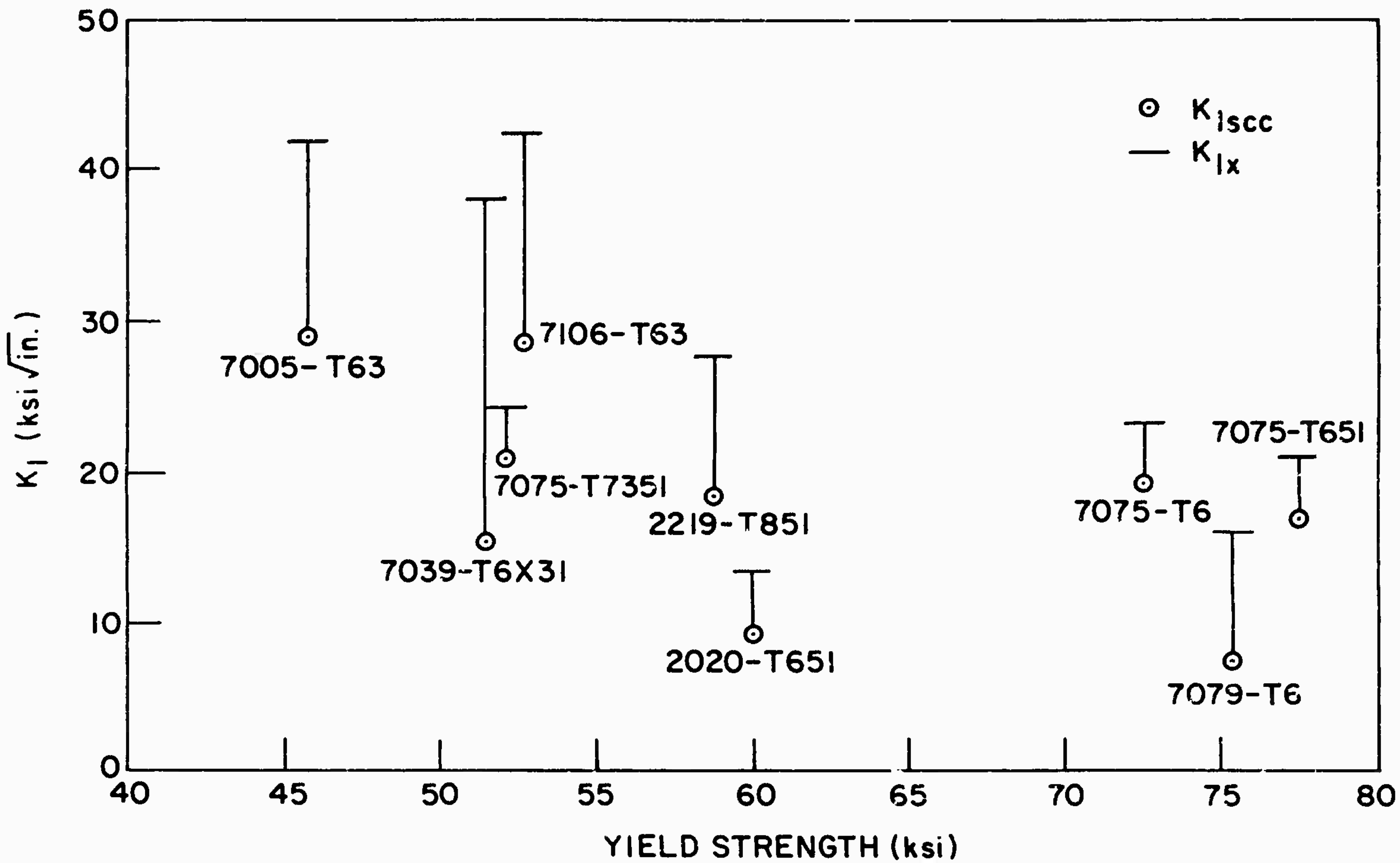

Fig. C-1 - Stress corrosion cracking resistance index

D. ABSTRACTS OF MANUSCRIPTS AND REPORTS

1. H. W. Paxton and R. P. M. Procter, "The Effects of Machining and Grinding on the Stress-Corrosion Cracking Susceptibility of Metals and Alloys," Technical Paper EM 68-520, American Society of Tool and Manufacturing Engineers, Symposium on Surface Integrity in Machining and Grinding, Pittsburgh, Pa., January 24-25, 1968

A brief introduction to the phenomenology of stress-corrosion cracking in metals and alloys is provided; the practical importance and dangers of the phenomenon and its wide-spread nature are emphasized. The changes produced in the surface layers of metals by machining and grinding operations, which can affect stress-corrosion susceptibility, are discussed. Published data on the effect of machining and grinding on the stress-corrosion susceptibility of the major alloy systems (brasses, carbon steels, high-strength steels, stainless steels, aluminum alloys and titanium alloys) are reviewed. Some general recommendations and suggestions for future research are made.

2. N. M. Lowry, O. R. Mulkey, J. M. Kuronen, and J. W. Bieber, "A Method of Measuring Crack Propagation Rates in Brittle Materials," Document D6-60072, The Boeing Company, May 1967.

A method of measuring the rate of crack propagation is described. The method, applicable to any specimen having a smooth surface across which brittle-type fracture will occur, is accomplished by means of a fine gridwork of conducting lines applied to the specimen by various vacuum deposition techniques. When a potential is applied to the grid, a direct reading of current (or distance) versus time is obtained with a strip chart recorder or equivalent device. The method has been applied to the study of brittle-type fracture associated with stress-corrosion cracking observed in titanium alloys.

3. H. R. Smith, D. E. Piper, and F. K. Downey, "A Study of Stress-Corrosion Cracking by Wedge-Force Loading," Document D6-19768, The Boeing Company, June 1967.

Wedge-force loading a center-cracked sheet specimen provides a unique resolution of whether stress-corrosion cracking (SCC) depends on an applied crack-tip stress-intensity factor or on net-section stresses. With this loading, crack extension causes a decreasing stress-intensity factor at the crack tip, while the net-section stresses increase. Therefore, when stress-corrosion crack growth is arrested in this specimen, the dependency on stress intensity is proved. The stress intensity at arrest agrees remarkably well with K_{SCC} values determined by more conventional techniques that establish crack initiation thresholds through multiple tests. Crack growth rate data important to the establishment of inspection intervals in structures can also be obtained. Finally, the technique affords considerable economy of test time and material costs in stress-corrosion studies.

4. J. C. Williams, "Some Observations on the Stress-Corrosion Cracking of Three Commercial Titanium Alloys," Document D6-19553, The Boeing Company, September 1967.

Stress-corrosion cracking has been studied in commercial titanium alloys Ti-8Al-1Mo-1V, Ti-6Al-4V, and Ti-4Al-3Mo-1V. Electron fractography has been used in conjunction with electron metallography to establish the fracture path. A sharp transition from ductile to cleavage failure occurs in the alpha phase between 4 and 6 weight percent aluminum, while the beta phase fails by ductile rupture in all cases. Thin-foil electron microscopy has been used to show that the dislocation arrangements produced in the alpha phase range from tangles in the 4 weight percent aluminum alloy to coplanar arrays in the 6 and 8 weight percent alloys. The relation between dislocation arrangements and fracture mode offers a qualitative explanation of the variations in environmental susceptibility of various titanium alloys.

5. D. N. Fager and W. F. Spurr, "Some Characteristics of Aqueous Stress Corrosion in Titanium Alloys," Document D6-60083, The Boeing Company, September 1967.

The influence of microstructural features on the stress-corrosion fracture path has been studied in the alloys Ti-8Al-1Mo-1V, Ti-6Al-4V, Ti-4Al-3Mo-1V, Ti-5Al-2.5Sn, and Ti-13V-11Cr-3Al by means of optical microscopy, electron microscopy, and X-ray diffraction. These studies have shown that susceptibility of titanium alloys to aqueous stress corrosion is influenced by crystalline structure of the susceptible phase (bcc or hcp), preferred grain orientation, and relative phase content where one phase is immune. Stress-corrosion cracking occurs on or near the {100} planes in the bcc beta phase, but can only occur near the single (001) plane in the hcp alpha phase. This restriction of cracking in the alpha phase results in a significant influence due to preferred orientation; it also contributes to the influence of stress state on susceptibility in the high-alpha alloys. The apparent lack of correlation between the structures of the phases and their stress-corrosion susceptibilities indicates that the basic mechanism is surface-controlled rather than structurally controlled.

6. M. J. Blackburn and J. C. Williams, "Metallurgical Aspects of the Stress-Corrosion Cracking of Titanium Alloys," presented at "Fundamental Aspects of Stress-Corrosion Cracking," Ohio State University, September 11-15, 1967; to be published in Proceedings

This paper should be read in conjunction with that of T. R. Beck*, which deals with the electrochemical factors influencing the stress-corrosion cracking (SCC) of titanium alloys in aqueous solutions.

* "Electrochemical Aspects of Titanium Stress-Corrosion Cracking" by T. R. Beck. Presented at "Fundamental Aspects of Stress-Corrosion Cracking" at Ohio State University, September 11-15, 1967.

The metallurgical and mechanical factors which obviously interact with the electrochemistry are considered here. The number of variables make generalization difficult unless these are qualified by specification of the test conditions. It is shown that failure of α-phase in Ti-Al alloys and in the commercial alloy Ti-8Al-1Mo-1V under SCC conditions occurs by cleavage on the $\{10\bar{1}8\}$ - $\{10\bar{1}7\}$ plane of the hexagonal phase. The general phase structure of these alloys is described and it is shown that the $\alpha \rightarrow \alpha + \alpha_2$ (Ti_3Al) transformation is of considerable importance in determining susceptibility. In the commercial alloy, presence of the β-phase and the martensitic phases reduces susceptibility. Results on the binary Ti-Al alloys indicate that the onset of susceptibility to stress-corrosion cracking occurs between 4-5% aluminum which correlates with a change from homogeneous to planar dislocation arrangements. The influence of stress on the nucleation and propagation of a crack has been analyzed for precracked specimens, using the methods of linear elastic mechanics. From these results, several factors which influence the nucleation stage are discussed. It is proposed that for a propagating crack, there are two components of fracture, i.e., electrochemical and mechanical. At low α-phase strength levels or aluminum contents the electrochemical component predominates, while at higher strength levels or aluminum content the mechanical component increases. Some qualitative correlations are made between mechanical properties, deformation structures, fracture mode, and susceptibility to stress-corrosion cracking.

7. R. W. Huber, R. J. Goode, and R. W. Judy, Jr., "Fracture Toughness and Stress-Corrosion Cracking of Some Titanium Alloy Weldments," Welding Journal Research Supplement, October 1967, pp. 1-9.

Fracture toughness and stress-corrosion cracking characteristics have been determined for a variety of 1 in. thick titanium alloy plates and welds. The drop-weight tear test (DWTT) and a bend-bar K_{Ic} test were used in the fracture toughness studies. A precracked cantilever bend specimen was used for the stress-corrosion cracking studies.

E. TITLES OF PREVIOUS REPORTS

1. Matthew Creager, "The Elastic Stress Field Near the Tip of a Blunt Crack," (Master's Thesis) Lehigh University October 1966

2. E. P. Dahlberg, "An Annotated Bibliography of Recent Papers and Reports on the Subject of Ambient Temperature Aqueous Stress-Corrosion Cracking of Titanium and Titanium Alloys," NRL Bibliography Report 29, October 1966

3. E. P. Dahlberg (General Editor), "ARPA Coupling Program on Corrosion (First Quarterly Report)," NRL Memorandum Report 1739, December 1966

4. G. Sandoz and R. L. Newbegin, "Stress-Corrosion Cracking Resistance of an 18Ni 200 Grade Maraging Steel Base Plate and Weld," NRL Memorandum Report 1772, March 1967

5. G. Sandoz, "Effects of Some Organics on the Stress-Corrosion Susceptibility of Some Titanium Alloys," article in DMIC Memorandum 228 (entitled "Accelerated Crack Propagation of Titanium by Methanol, Halogenated Hydrocarbons, and other Solutions"), Battelle Memorial Institute, March 6, 1967

6. George Sandoz and R. L. Newbegin, "Stress and Corrosive Environments (Some Environmental Effects on Titanium Alloys)," Report of NRL Progress, March 1967, pp. 28-30

7. G. Sandoz, "Stress-Corrosion Cracking Susceptibility of a Titanium Alloy in a Non-electrolyte," Nature, Vol. 214, April 8, 1967, pp. 166-167

8. E. P. Dahlberg (General Editor), "ARPA Coupling Program on Stress-Corrosion Cracking (Second Quarterly Report)," NRL Memorandum Report 1775, April 1967

9. A. M. Sullivan, "Dissolution Velocities of Different Organic Media," Report on NRL Progress, April 1967, pp. 18-19

10. E. P. Dahlberg, "Thin Foil Electron Microscopy," Report of NRL Progress, April 1967, pp. 19-21

11. G. Sandoz, "Delayed Fracture Characteristics of Ti-6Al-1Mo-1V Alloy," Report of NRL Progress, May 1967, pp. 31-32

12. N. M. Lowry, O. R. Mulkey, J. M. Kuronen, and J. W. Bieber, "A Method of Measuring Crack Propagation Rates in Brittle Materials," Document D6-60072, The Boeing Company, May 1967

13. C. O. Timmons, R. L. Patterson, and L. B. Lockhart, Jr., "The Adsorption of C-41 Labeled Stearic Acid on Iron," NRL Report 6553, June 2, 1967

14. H. R. Smith, D. E. Piper and F. K. Downey, "A Study of Stress-Corrosion Cracking by Wedge-Force Loading," Document D6-19768, The Boeing Company, June 1967

15. R. W. Judy, Jr. and R. J. Goode, "Stress-Corrosion Cracking Characteristics of Alloys of Titanium in Salt Water," NRL Report 6564, July 21, 1967

16. E. P. Dahlberg (General Editor), "ARPA Coupling Program on Stress-Corrosion Cracking (Third Quarterly)," NRL Memorandum Report 1812, August 1967

17. D. A. Meyn, "Effect of Crack Tip Stress Intensity on the Mechanism of Stress-Corrosion Cracking of Titanium-6Al-4V in Methanol," Corrosion Science, Vol. 7, No. 10, October 1967, pp. 721-723

18. M. Creager and P. Paris, "Elastic Equations for Blunt Cracks with Reference to Stress Corrosion Cracking," International Journal of Fracture Mechanics, Vol. 3, No. 4, December 1967, pp. 247-252

19. E. P. Dahlberg (General Editor), "ARPA Coupling Program on Stress-Corrosion Cracking (Fourth Quarterly," NRL Memorandum Report 1834,November 1967

20. R. P. Wei, "Fracture-Crack Propagation in a High-Strength Aluminum Alloy," to be published in Journal of Fracture Mechanics

21. R. P. Wei, "Application of Fracture Mechanics to Stress Corrosion Studies," to be published in Proceedings - International Conference on Corrosion, Ohio State, September 1967

22. G. W. Graves and D. A. Shockey, "Effect of Water on the Toughness of MgO Crystals," submitted to Journal of American Ceramic Society

23. George Sandoz, "Subcritical Crack Propagation in Ti-8Al-1Mo-1V Alloy in Organic Environments, Salt Water, and Inert Environments," to be published in Proceedings - International Conference on Corrosion, Ohio State, September 1967

24. B. F. Brown, "The Fracture Mechanics of Stress-Corrosion Cracking," submitted to Metallurgical Reviews

F. ABSTRACTS OF SOME RECENT ARTICLES ON STRESS-CORROSION CRACKING

1. G. J. Petrak, "Dynamic Subcritical Crack Growth Properties of Duplex Annealed Ti-8Al-1Mo-1V and Mill Annealed Ti-6Al-4V in an Air and Corrosive Environment," Technical Report AFML-TR-66-392, Wright-Patterson Air Force Base, Ohio, January 1967

This program was conducted to determine the dynamic (fatigue) crack growth properties of two titanium alloys (Ti-8Al-1Mo-1V Duplex Annealed and Ti-6Al-4V Mill Annealed) at room temperature in an air and in a 3.5 percent NaCl environment. Dynamic crack growth versus cycles to failure was determined at two loading frequencies (40 cpm and 2 cpm) for the corrosive environment and at a 40 cpm loading frequency for the air environment. A comparison of the air and corrosive environment test data at the 40 cpm loading frequency shows a reduction in cyclic life when exposed to the 3.5 percent NaCl environment. Also a comparison of the corrosive environment test data at loading frequencies of 40 cpm and 2 cpm shows a reduction in cyclic life on both a time and a number of cycles to failure basis at the higher loading frequency.

2. W. A. Mannehimer and H. W. Paxton, "Some Effects of Nitrogen on the Resistance to Stress Corrosion Cracking of Type 304 Stainless Steel Wires," Contract Nonr 760(14) NR 036-C29, May 1, 1967, Office of Naval Research

This investigation dealt with A. Nature and morphology of surface layer formed in nitrided type 304 steel; B. Study of stress corrosion cracking resistance of nitrided type 304 steel; C. Electrochemical and metallographic observations on stress corrosion cracking of nitrided type 304 steel; and D. Diverse supporting experiments including the mechanical behavior of nitrided wire and intergranular and generalized corrosion.

3. J. H. Terrell, R. H. Forsyth, and C. S. Naiman, "Studies of Stress Corrosion Cracking by the Mossbauer Effect," Mithras, Incorporated, Cambridge, Massachusetts. Office of Naval Research, MC 66-133C-R1, Final Technical Report, Contract No. N00014-67-C-0483, July 31, 1967

An investigation into various techniques for carrying out Mossbauer-Effect (ME) measurements in a nondestructive in-situ manner requiring no sample preparation (backscatter ME measurements) is reported. The research was directed toward detecting the 2 percent abundant Fe^{57} nucleus which is common to iron alloys so that environmental changes brought about by stress and/or corrosion could be followed. Comparisons of three backscatter-ME experiments which employ different geometrical arrangements of the source, sample, and counter, and which detect the 14.4-kev γ-rays or 6.3-kev internal conversion X-rays, are given. It is found that the most efficient technique for performing backscatter-ME measurements is to use a geometrical arrangement designated as "around-the-corner" detecting 6.3-kev internal conversion X-rays.

4. S. B. Brummer, R. O. Bell, and F. H. Cocks, "Study of the General Mechanism of Stress Corrosion of Aluminum Alloys and Development of Techniques for Its Detection," Tyco Laboratories, Incorporated, Bear Hill, Waltham, Massachusetts. NASA, Quarterly Report No. 4, Contract No. NAS 8-20297, August 31, 1967

The mechanism of the stress-corrosion cracking of high-strength aluminum alloys has been investigated using electrochemical, mechanical, and electron-microscopic techniques. Experiments have been carried out in 1 M NaCl buffered at pH 4.7 at 30 C, using the commercial alloys 7075 and 2219 and relevant pure materials. The feasibility of detecting stress-corrosion damage in fabricated aluminum-alloy parts by nondestructive-testing techniques has been investigated using ultrasonic surface waves (Rayleigh waves).

5. A. J. Jacobs, "The Effects of Point Defects and Dislocations on the Stress-Corrosion Susceptibility of Aluminum Alloys," North American Rockwell Corporation, Rocketdyne Division, Canoga Park, California. NASA, George C. Marshall Space Flight Center, R-7244, Quarterly Report, Contract No. NAS 8-20471, September 30, 1967

The requisite 7075 aluminum-alloy test material has been received and heat treated to the -T6 and -T73 tempers, and subsequently machined and characterized. The initial stress-corrosion tests on 7075-T6 and 7075-T73 control specimens have been highly successful; continuous monitoring of the test specimens by means of an ultrasonics technique has revealed a very early stage in an overall two-stage cracking process. Supersaturated concentrations of vacancies have been introduced into 7075 by heating at elevated temperatures in the range from 750 to 895 F and then quenching in iced brine. The aging curves that were subsequently obtained showed the expected dependence on quenching temperature. Attempts have been made to grow single crystals of 7075 by means of a modified Bridgman method and a strain-anneal method.

6. W. J. Helfrich, "Development of a Rapid Stress-Corrosion Test for Aluminum Alloys," Kaiser Aluminum & Chemical Corporation, Spokane, Washington. NASA, Quarterly Progress Report No. 6, Contract No. NAS 8-20285, September 15, 1967

Short-transverse stress-corrosion cracking of alloys 2024, 2014, 2219, 7075, 7079, and 7039 was studied in various laboratory and natural environments. These included continuous immersion in a 1 percent NaCl-2 percent $K_2Cr_2O_7$ solution at 60 C and boiling 0.5 percent NaCl-2 percent $K_2Cr_2O_7$ solution, alternate immersion in 3.5 percent NaCl solution, and outdoor exposures at Daytona Beach, Florida (marine environment) and Pittsburgh, Pennsylvania (industrial environment). The majority of these tests were performed on C-ring specimens, but some stress-corrosion data have been obtained in

3.5 percent NaCl alternate immersion tests using 1/8-inch-diameter tensile rounds. The results of these studies are discussed in terms of the general applicability of laboratory tests in (1) separating alloys and tempers which exhibit a range of susceptibility to stress-corrosion cracking and (2) simulating the stress-corrosion performance of 2000- and 7000-series alloys in natural environments.

7. T. L. Mackay and N. A. Tiner, "Stress Corrosion Cracking of Titanium Alloys at Ambient Temperature in Aqueous Solutions," Douglas Aircraft Company, Incorporated, Newport Beach, California. NASA, SM-49105-Q4, Quarterly Progress Report, Contract No. NAS 7-488, October 1967

The effect of microstructure on stress-corrosion cracking of Ti-8Al-1Mo-1V was studied by solution heat treatment at 1600 F, 1800 F, and 2000 F. Amounts of hydrogen from 27 to 100 ppm were introduced at the above temperatures. Ti-8Al-1Mo-1V, with solution heat treatments at 1800 F and 2000 F followed by air quenching, were immune to stress-corrosion cracking in 3 percent salt solution. Ti-8Al-1Mo-1V, with solution heat treatment at 1600 F, was susceptible to stress-corrosion cracking at these hydrogen concentrations.

Transmission-electron microscopic studies were made on Ti-8Al-1Mo-1V, Ti-6Al-4V, and Ti-5Al-2.5Sn. Thin foils were immersed in 5 percent NaCl solution and stressed by bending. No evidence of any local anodic sites could be detected by this technique.

8. W. Beck, E. J. Jankowsky, and W. H. Golding, "Fatigue and Delayed Brittle Failure of Vacuum Melted and Cadmium Plated Steel," Corrosion Science, Vol. 7, No. 10, October 1967, p. 709

The detrimental effect of cyanide cadmium plating on fatigue strength and delayed brittle failure performance of an ultra high strength steel is reduced effectively by using vacuum melted steel as the basis metal.

This phenomenon has been related predominantly to the increased tensile ductility of the steel with comparatively low hydrogen concentration.

9. B. W. Lifka, D. O. Sprowls, and J. G. Kaufman, "Exfoliation and Stress-Corrosion Characteristics of High Strength, Heat Treatable Aluminum Alloy Plate," Corrosion, Vol. 23, No. 11, November 1967, p. 335

Corrosion tests in laboratory accelerated environments and in seacoast and inland industrial atmospheres have been conducted on specimens from 1 3/8 in. thick hot rolled plate of several, high strength, heat treatable, aluminum alloys and tempers.

10. S. M. Toy, "Polarization of Strained Titanium Alloys in Sodium Chloride Solutions," Douglas Aircraft Company, Newport Beach, California. Douglas Paper 4458, August 1967

The electrode reactions involved in the stress corrosion of Ti alloys in sodium chloride solutions were diagnosed from potentiostatic experiments. The redox cell system involves two principle cathodic reactions. The anodic reactions were complex. The electrochemical reactions are affected by the application of stress, by oxygen content, and the heat treated condition of the materials. The effect of stress is to shift the polarization curves in an electronegative direction whereas the effect of oxygen shifts the polarization curves in the electropositive direction. The corrosion current for strained Ti-6Al-4V was found to be higher than that for strained Ti-5Al-2.5Sn.

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G. DIARY OF EVENTS

The ARPA Coupling Program on Stress-Corrosion Cracking fall quarterly meeting was held at NRL on 5 December 1967. The one-day meeting was devoted to a review of current work on iron and steels and included presentations from both ARPA and non-ARPA personnel.

An ad hoc two-day ARPA Program Review was held at NRL on 11-12 December 1967 for ARPA management personnel. Principal aspects of the coupling program were covered in separate sessions devoted to surface physics, surface chemistry, test methods, titanium, steels, aluminum and nonmetallics.

Project personnel took part in the ASTM technical subcommittee groups of Committee G-1 dealing with stress-corrosion cracking at Atlantic City on 29 January - 1 February 1968.

The winter quarterly meeting was held at The Boeing Company, Seattle, Washington, on 12-13 February 1968. The meeting was devoted to a review of the physical metallurgy and stress-corrosion cracking of titanium alloys.

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13. ABSTRACT

A progress report of the research investigations being carried out on the problem of stress-corrosion cracking of high strength materials under ARPA Order 878 is presented. Work at Carnegie-Mellon University, Lehigh University, Georgia Institute of Technology, The Boeing Company, and the Naval Research Laboratory concerning physical metallurgy, surface chemistry, fracture mechanics, and characterization tests and translation related to stress-corrosion cracking is described. The materials being studied include high strength steels, titanium alloys, and aluminum alloys. Abstracts of recently published reports and a diary of events are included.

14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Stress-corrosion cracking						
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Surface chemistry						